

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2355

ACHROMATIZATION OF DEBYE-SCHERRER LINES

By Hans Ekstein and Stanley Siegel

Armour Research Foundation



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SUMMARY

A method is described for reducing the width of a Debye-Scherrer line produced by diffraction from a polycrystalline medium, if this width is due to the spectral impurity of the primary characteristic radiation.

In this method, a diverging polychromatic beam is allowed to fall on the plane surface of a single crystal. The beam diffracted by this crystal will diverge and will contain a bundle of rays whose wave-length range corresponds to the finite spectral width of the characteristic radiation. The polycrystalline sample is mounted normal to this bundle. It is shown that the different wave lengths diffracted by the sample can be brought to a narrow focal spot.

INTRODUCTION

It has been shown (reference 1) that the chief factor in limiting the precision of stress measurements by X-rays is the spectral width of the primary characteristic radiation. The diffracted line has a width dependent upon the angle of diffraction. At large diffracting angles where the line is most sensitive to lattice dimension changes, the spectral width will predominate over that width produced by the geometry of the collimating system, provided that the collimator size is sufficiently small. The limit of precision of stress determinations is given essentially by the accuracy with which the center of such a line can be measured. It was further shown that a smooth line can only be produced under those favorable conditions in which the crystal statistics are correct, otherwise an irregular diffraction line is produced. Even if a smooth intensity distribution is recorded by the photographic method, an error in measurement is present because of the irregular fluctuations of density caused by the film grain.

¹A brief summary of this work appeared in "Letters to the Editor," Phys. Rev., vol. 73, no. 10, second ser., May 15, 1948, pp. 1207-1208.

A sharpening of the diffraction line by monochromatization of the primary or secondary beam can be achieved by utilizing a double crystal spectrometer, but the loss of intensity to be expected from such an arrangement would be serious. However, it does appear that it is possible to bring rays of different wave lengths to an intersection after diffraction. This procedure, which is termed "achromatization," seems likely to succeed in narrowing the diffraction line without a prohibitive loss in intensity. It appears that by the use of achromatization the precision of lattice-parameter measurements for stress determinations can be increased by an order of magnitude. An investigation of this method, conducted at the Armour Research Foundation, was sponsored by and conducted with the financial assistance of the National Advisory Committee for Aeronautics.

The authors wish to thank Mr. A. Guinier for an inspiring discussion.

ACHROMATIZATION WITH A POINT SOURCE

In geometrical optics, a divergent beam is brought to a focus by lenses and mirrors. A similar result can be obtained for monochromatic X-rays by the use of crystals as reflectors (Bragg-Brentano focusing). The problem of focusing rays of different color (achromatization) is solved in geometrical optics by the use of lenses of different indices of refraction. X-rays focused in the usual way (geometrically) exhibit "chromatic aberration." It will be shown that achromatization of X-rays can be achieved, but at the expense of intensity and in such a manner that at a certain point only one wave length of every ray within a divergent bundle is used.

In figure 1, a beam of X-rays diverges from the point source of X-rays at A. This diverging beam, composed of rays of different wave lengths, strikes a surface QT of a crystal monochromator QTUR. The surface QT makes an angle α with the normal to a prominent diffracting plane, namely, dd' of interatomic distance d_m . The monochromator is set to diffract the most intense wave length of the characteristic radiation at some angle θ_m , the angle θ_m being defined by the central ray AC. After diffraction by the monochromator, the beam diverges onto the polycrystalline sample P.

The central ray will diffract at the angle θ_m and the diffracted ray CE will have a wave length λ . The ray AB strikes the monochromator at an angle $\theta_m + d\theta_m$, and the diffracted ray BH will have a wave length $\lambda + d\lambda$. The beam diffracted by the monochromator can be considered to arise from an apparent point source A'.

The polycrystalline sample is adjusted so that the central ray A'E is normal to its surface. Among the many atomic planes in the sample, consider one, whose spacing is d , which diffracts the central ray of wave length λ at an angle θ . It is apparent that a circular cone of X-rays of semiapex angle $180^\circ - 2\theta$ is diffracted by the sample, but for purposes of the analysis only rays in the plane of ACE are considered.

The ray A'E strikes a plane of spacing d at an angle θ , and the diffracted ray will travel in the direction EN. In the same manner, the ray A'H of wave length $\lambda + d\lambda$ requires that the plane of spacing d be oriented at an angle $\theta + d\theta$ if there is to be diffraction at H. The angle between the diffracted ray HN and the incident ray A'H has now decreased to $180^\circ - 2(\theta + d\theta)$. The distance $EN = F$ defines the point where the rays EN and HN intersect.

Consider the ray ABH diffracted by the monochromator. Differentiation of Bragg's law gives:

$$d\lambda = 2 d_m \cos \theta_m d\theta_m \quad (1)$$

and, for the same ray, after second diffraction at H:

$$d\lambda = 2 d \cos \theta d\theta \quad (2)$$

Therefore,

$$d_m \cos \theta_m d\theta_m = d \cos \theta d\theta$$

or

$$d\theta = \frac{d_m \cos \theta_m}{d \cos \theta} d\theta_m \quad (3)$$

But

$$\gamma = 2 d\theta + d\theta_m \quad (4)$$

and, if $A'E = L$ and $EN = F$, then from figure 1,

$$- \frac{F\gamma}{\cos 2\theta} = L d\theta_m \quad (5)$$

Combining equations (3) to (5),

$$\frac{L}{F} = \left(1 + \frac{2 d_m \cos \theta_m}{d \cos \theta} \right) \left(- \frac{1}{\cos 2\theta} \right) \quad (6)$$

Since

$$\frac{d_m}{d} = \frac{\sin \theta}{\sin \theta_m}$$

equation (6) reduces to

$$\frac{L}{F} = \left(1 + \frac{2 \tan \theta}{\tan \theta_m}\right) \left(-\frac{1}{\cos 2\theta}\right) \quad (7)$$

which is the condition for achromatization.

It is apparent from figure 1 that the distance $L = A'E$ cannot be measured directly. However, L may be written as

$$L = l' + V \quad (8)$$

where $l' = A'C$ and $V = CE$. The distance V may be measured directly and l' may be obtained in terms of l (equal to AC) which may also be measured directly.

From figure 1,

$$\frac{CJ}{CK} = \frac{l'}{l} \quad (9)$$

but

$$\left. \begin{aligned} \angle JBC &= \theta_m + \alpha - 90^\circ \\ \angle KCB &= \theta_m - \alpha \end{aligned} \right\} \quad (10)$$

Thus

$$\left. \begin{aligned} CJ &= -BC \cos (\theta_m + \alpha) \\ CK &= BC \cos (\theta_m - \alpha) \end{aligned} \right\} \quad (11)$$

and

$$\frac{l'}{l} = -\frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)} \quad (12)$$

Combining equations (8) and (12),

$$L = -l \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)} + V \quad (13)$$

and equation (7) may now be written

$$V = F \left(1 + \frac{2 \tan \theta}{\tan \theta_m}\right) \left(-\frac{1}{\cos 2\theta}\right) + l \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)} \quad (14)$$

Equation (14) expresses the distance V between monochromator and sample in terms of measurable quantities.

In present X-ray tubes, the focal area on the target is so large that the condition that A be a point source is not even approximately realized. Even recent advances in focusing of the electron beam do not provide an X-ray source small enough to be considered a point or a line source. On the other hand, it is possible to choose α such that an extended source may be effectively reduced in size. It is clear from the derivation that the above equations are valid only for small deviations $d\lambda$. The actual purpose of the method is to achromatize a single characteristic line. In this case, $d\lambda/\lambda$ is of the order of 10^{-4} so that the approximation is justified.

ACHROMATIZATION WITH AN EXTENDED SOURCE

The purpose of this section is to consider a system which projects an achromatized and reduced image of the source upon a film. The method used for reduction is similar to one treated by Brentano (reference 2), Stephen and Barnes (reference 3), and Fankuchen (reference 4).

In figure 2, $ACEN$ is the path of the central ray (of wave length λ). If the source is of extent S , one may consider another central ray $A_1C'E'N'$ of wave length λ , necessarily parallel to the first, which will reach a point E' separated from E by a distance S' on the sample. After diffraction, the second ray reaches a point N' separated by a distance S'' from N . By a proper choice of α , S'' can be made to be very small.

In figure 2,

$$\left. \begin{aligned} \angle JCC' &= \theta_m + \alpha - 90^\circ \\ \angle CC'K &= \theta_m - \alpha \end{aligned} \right\} \quad (15)$$

hence,

$$S' = -S \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)} \quad (16)$$

However, the angle between the rays $C'E'$ and $E'N'$ is $180^\circ - 2\theta$; hence,

$$S'' = -S' \cos 2\theta \quad (17)$$

Combining equations (16) and (17),

$$S'' = S \cos 2\theta \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)} \quad (18)$$

In equation (18), $\theta_m + \alpha$ may be so chosen that the extended source S (object) can be reduced to a very small image, S". As an example, assume that the true width of the X-ray focal spot is 1 millimeter and that θ and θ_m are each 80° . If α is taken as 20° , then S" becomes 0.3 millimeter. On the other hand, if the source A were already very small, say 0.1 millimeter (produced by electron focusing), then with the above values it should be theoretically possible to reduce this source to an image only 0.03 millimeter in extent.

GEOMETRY OF THE APPARATUS

According to equation (18), $\theta_m + \alpha$ is to be chosen so that $\cos(\theta_m + \alpha)/\cos(\theta_m - \alpha)$ is very small; hence, in equation (14), V will show little dependence on l , and equation (14) may be written as

$$V \approx F \left(1 + \frac{2 \tan \theta}{\tan \theta_m} \right) \quad (19)$$

In precision measurements of lattice parameters it is necessary to use θ values as large as possible, preferably near 80° or more. According to equation (19), V will be comparable with F only if θ_m is approximately equal to θ . Thus, if $\theta_m = \theta$, and a film-to-specimen distance F of 5 centimeters is chosen, then V will be about 15 centimeters, a value which is of the same order of magnitude as the distances used in the conventional back-reflection technique. If θ_m is much smaller than θ , it is apparent that, for a fixed value of F, V will become quite large.

The above fact imposes a somewhat stringent condition upon the geometrical arrangement; it is practically necessary that the beam diffracted by the monochromator travel through the X-ray tube. This arrangement can be realized by the schematic drawing presented in figure 3. The jacket of the X-ray tube is J, and the source of diverging X-rays is at the target A. The crystal monochromator may be inside or immediately outside the X-ray tube, but, for maximum utilization of intensity, it is advisable that the monochromator be located within the tube. The beam diverging from the monochromator M after diffraction is then allowed to traverse the tube window W. The advantage of this arrangement is that the tube may be handled in the conventional manner and no size restrictions are imposed upon the sample P.

The position of the film can be obtained from equation (19). Since θ is close to 90° , let

$$\epsilon = 180^\circ - 2\theta \quad (20)$$

Then, from equation (19),

$$\frac{V}{F} \approx 1 + \frac{4}{\epsilon \tan \theta_m} \quad (21)$$

or

$$F \approx \frac{V \epsilon \tan \theta_m}{4 + \epsilon \tan \theta_m} \quad (22)$$

This curve behaves like a spiral for very small angles ϵ , but its slope decreases more than that of a spiral as θ approaches θ_m . A plot of F as a function of θ for a fixed value of V shows the trace ff' (fig. 3), which is the film position. It is apparent that, for slight changes of d , the position of the image N will change more rapidly than would be for the case for a diffraction line produced in the conventional back-reflection technique.

EXPERIMENTAL STUDY

A very simple experimental arrangement and procedure was considered for verifying the principle of achromatization. It would be desirable to attempt to produce a sharp line by the methods outlined, but such an experiment would be beset by many experimental difficulties which could not be undertaken for the present. A primary consideration for producing a sharp line would be the necessity for offsetting the very long (photographic) exposures by use of Geiger counters. This, however, must be the subject of future work.

A wide wave-length band may be obtained by reflecting the $K\alpha_1$ and $K\alpha_2$ components from a sodium-chloride crystal. The achromatization may be verified by bringing the $K\alpha_1$ and $K\alpha_2$ components into coincidence. This is a wider wave-length spread than would normally be obtained in usual diffraction effects; nevertheless, it is an excellent way for checking the achromatization principle.

In figure 1, it is seen that, if a film is mounted normal to the ray CE , a circular cone of rays diffracted by the sample P will in effect give rise to a circular diffraction line on the photographic film, but the line in the region near N will be narrow because of the achromatization of the beam. Each ray of wave length $\lambda \pm d\lambda$ diffracted by the sample P makes an angle of $180^\circ - 2(\theta \pm d\theta)$ with the corresponding incident ray; hence, a series of cones of semiapex angles of $180^\circ - 2(\theta \pm d\theta)$ are formed which have a common intersection at N , but diverge at any other point. In fact, the maximum divergence occurs at a point diametrically opposite N . Thus, with a film mounted normal to

the ray CE, it would be expected that the region near N should show some narrowing of the diffraction line. Of course, it is necessary that the sample be properly chosen so that no line widening as a result of small particle size will occur.

Figure 4 shows the experimental arrangement. A widely divergent bundle of rays from the target strikes the monochromator M at an angle θ_m . The tube T serves to limit the bundle and prevent excessive fluorescence from the camera parts. The film holder K is mounted normal to the beam diffracted by the monochromator. The monochromatized beam passes through an aperture O in the film holder onto the specimen holder G which is adjustable.

The focal spot area on the target of the tube used in these experiments was 10 millimeters by 1 millimeter, with the longer direction in the line of sight between the target and the monochromator. The apparatus was tilted so that the monochromator "viewed" the target at a very small angle, approximately $1\frac{10}{2}$, thus producing a small "apparent" focal spot, the size of this apparent focus being about 1 by 0.26 millimeter. The apparatus was oriented so that perfect achromatization of the rays diffracted by the polycrystalline sample would correspond to a line width of 0.26 millimeter. Thus, the source was treated as an approximate point source and a flat monochromator ($\alpha = 90^\circ$) was used. Under these conditions, $AC = A'C$ and $ACE = L$ (fig. 1) and equation (14) becomes:

$$\frac{L}{F} = \left(1 + \frac{2 \tan \theta}{\tan \theta_m} \right) \left(- \frac{1}{\cos 2\theta} \right) \quad (23)$$

which may be used.

A sample of iron was chosen for use as the polycrystalline medium. A preliminary study of the iron sample showed no line broadening as a result of small crystallite size but did show a number of reasonably large crystals which caused a "peppering" of spots on the Debye-Scherrer line. The sodium-chloride monochromator was set at approximately 72° to produce the $K\alpha_1$ and $K\alpha_2$ components of the (442) reflection with cobalt radiation, the setting being such that the $K\alpha_1$ was of a higher visual intensity than the $K\alpha_2$ (as seen on a fluorescent screen). Both beams, well-separated, were allowed to fall on the iron sample. Thus, two (310) reflections arose from the iron; one corresponding to the $K\alpha_1$ and the other to the $K\alpha_2$ component of cobalt. The angular spread of these lines was about 0.0134 radian which is about $2\frac{1}{2}$ times the width of the (310) reflection of iron produced in the conventional back-reflection technique. This width, of course, is greater than that of any line which normally would be used in precision determinations of lattice parameters, as most lines used for this purpose occur in the neighborhood of 80° .

The following constants were accordingly chosen:

$$\begin{aligned}\theta_m &= 72.2^\circ \\ \theta &= 80.8^\circ \\ F &= 7.92 \text{ centimeters}\end{aligned}$$

and, by equation (19), L becomes 39.6 centimeters.

Figure 5(a) shows the pattern obtained after a 100-hour exposure, the X-ray tube having been operated at 40 kilovolts and 7 milliamperes during this interval. The outer line is the $K\alpha_1$ component, the inner line is the $K\alpha_2$ component, and both coalesce quite uniformly at the focus N . The compression at the focus is quite appreciable, while diametrically opposite N the $K\alpha_1$ and $K\alpha_2$ lines show appreciable divergence.

The exposure time of 100 hours appears excessive. However, the original film is quite overexposed, and a reasonable photographic density in the region near N could have been obtained in probably one-quarter the time. Furthermore, the film may not have been at the exact focus.

A most striking observation can be made from figure 5(a); even with a heavy exposure, there are practically no reflections outside of the region of the focus. This contrasts sharply with the Debye-Scherrer photograph of the same sample (fig. 5(b)), in which the scatter of reflections outside of the Laue directions is quite pronounced.

The advantages to be gained by achromatization, beyond that gained from an expected improvement in the lattice parameter value, are quite apparent; large-grained materials could be expected to give reasonably sharp diffraction lines.

ESTIMATE OF INCREASE IN ACCURACY

It is possible to arrive at an order-of-magnitude value for the expected increase in accuracy of the lattice parameter when the diffraction line is achromatized. In the following analysis, it is assumed that in the conventional method the diffraction-line width is caused by the spectral impurity only, whereas the finite width of the source is taken into account for achromatization. Thus, the comparison is very favorable with the conventional method.

The absolute accuracy of a measurement depends on many factors, such as film grain size, experience of a visual observer, and so forth, which have been discussed previously. For purposes of comparison, however,

there need only be considered the resolution, as defined by a parameter difference $(\Delta d)_R$, such that two diffraction lines diffracted from atomic planes of spacings d and $d + \Delta d$, respectively, cannot be resolved if $\Delta d < (\Delta d)_R$. According to Rayleigh, two diffraction lines are considered resolved when the intensity of the minimum between the two line maximums is about 80 percent of the intensity of one of the maximums, provided the lines are of equal intensity.

In figure 6, ab represents the central ray of a diffraction line diffracted from a plane of spacing d in the sample. For a change in spacing from d to $d + \Delta d$, the central ray of a line is angularly displaced by an amount $2 \Delta \theta$ from the original line. On the film, the lines are separated by an amount Δl . If F is the distance between film and specimen,

$$\Delta \theta = \frac{\Delta l}{2F} \quad (24)$$

Figure 7 is a schematic representation of the intensity distributions of the two lines on the film. The abscissa x represents the distance along the film, and the point $x = 0$ corresponds to the position of one intensity maximum. The linear width of the diffraction lines at half maximum intensity is B .

The intensity distribution for a line may now be written as

$$I(x) = \frac{1}{x^2 + (B/2)^2} \quad (25)$$

and the peak intensity as

$$I(0) = \frac{1}{(B/2)^2} \quad (26)$$

If the resultant intensity of the minimum between the two lines is to be 80 percent of the maximum intensity, the value of x corresponding to an intensity of 40 percent $I(0)$ may be readily calculated. Thus,

$$I(x) = \frac{1}{x^2 + (B/2)^2} = \frac{4}{10} \frac{1}{(B/2)^2}$$

from which

$$x = \sqrt{\frac{3}{2}} \frac{B}{2} \quad (27)$$

Now

$$\Delta l = 2x = \sqrt{\frac{3}{2}} B \quad (28)$$

and substituting equation (28) into equation (24),

$$\Delta \theta = \sqrt{\frac{3}{2}} \frac{B}{2F} \quad (29)$$

The reciprocal resolving power $\left| \frac{\Delta d}{d} \right|_R$ is obtained by logarithmic differentiation of Bragg's law. Thus,

$$\left| \frac{\Delta d}{d} \right|_R = \left| \cot \theta \Delta \theta \right| \quad (30)$$

By substituting equation (29) into equation (30),

$$\left| \frac{\Delta d}{d} \right|_R = \left| \frac{\sqrt{\frac{3}{2}} \cot \theta B}{2F} \right| \quad (31)$$

The resolution for the two cases may now be calculated.

Debye-Scherrer lines.- In the case of Debye-Scherrer lines, the linear width B is

$$B = FW \quad (32)$$

where W is the angular width, given by

$$W = \frac{2w}{\lambda} \tan \theta_B \quad (33)$$

where w is the natural width of the incident characteristic radiation, λ is the wave length, and θ_B is the Bragg angle for the diffraction line. Substituting equation (33) into equation (31),

$$\left| \frac{\Delta d}{d} \right|_R = 1.22 \frac{w}{\lambda} \approx 5 \times 10^{-4}$$

The ratio w/λ is nearly constant for most wave lengths and is approximately 4×10^{-4} . The above expression is, of course, independent of the Bragg angle. Thus, as the distance between the sample and the film increases or the Bragg angle increases, the separation Δl will increase, but at the same time the width B will increase so that the ratio $\Delta l/B$ is fixed.

Achromatized lines.- In the case of achromatized lines, the width would be given by equation (18), that is,

$$B = S \cos 2\theta \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)}$$

Substituting this into equation (31),

$$\left| \frac{\Delta d}{d} \right|_R = \left| \frac{\sqrt{\frac{3}{2}} \cot \theta}{2F} S \cos 2\theta \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)} \right|$$

In order to evaluate this, it is necessary to assume reasonable values for the various quantities on the basis of conventional distances and angles used in precision lattice measurements. Furthermore, the focal spot size S is taken as 1/100 centimeter. This value is based on an X-ray tube now available.² Thus, with

$$\begin{aligned} \theta &= 80^\circ \\ F &= 15 \text{ centimeters} \\ S &= \frac{1}{100} \text{ centimeter} \end{aligned}$$

then

$$\left| \frac{\Delta d}{d} \right|_R \approx 7 \times 10^{-5} \frac{\cos (\theta_m + \alpha)}{\cos (\theta_m - \alpha)}$$

It should be noted that the expected error $\left| \frac{\Delta d}{d} \right|$ is about 5 to 15 times smaller than $\left| \frac{\Delta d}{d} \right|_R$, depending upon methods of observation. This has been discussed in a previous report (reference 1).

If the compression factor $\cos (\theta_m + \alpha) / \cos (\theta_m - \alpha)$ is unity, then under the above conditions an increase in accuracy by an order of magnitude may be expected, provided that the source is faithfully reproduced as an image. It is expected that a further increase in accuracy may be gained by cutting the crystal in the desired manner to reduce the compression factor. Finally, the Bragg angle may be increased and the film-to-specimen distance made larger; both factors will contribute to an increase in accuracy, since the diffraction line can always be focused.

²Designed by R. Pepinsky at the Alabama Polytechnic Institute, Auburn, Ala.

CONCLUSIONS

It has been shown that it is possible to achromatize an X-ray beam under proper geometrical conditions. All rays which issue from one source point can be made to intersect at one point after diffraction by a polycrystalline sample. The sharpness of the focal spot is limited by the size of the X-ray tube focus and the perfection of the crystal monochromator. A method has been described for effectively utilizing a broad X-ray focal spot and bringing rays from this large area to a narrow focus.

The new method is compared with the conventional one with respect to resolution of lines belonging to two nearby lattice parameters. Whereas the resolution of the conventional method is limited even for an ideal geometry, the resolution with achromatization can be made as large as is desired by decreasing the source width or increasing the film-to-specimen distance. Practically, an increase of resolution (and, therefore, of accuracy) of the order of 10 can be expected over the ideal optimum resolution of the conventional method.

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Chicago, Ill., June 1, 1948

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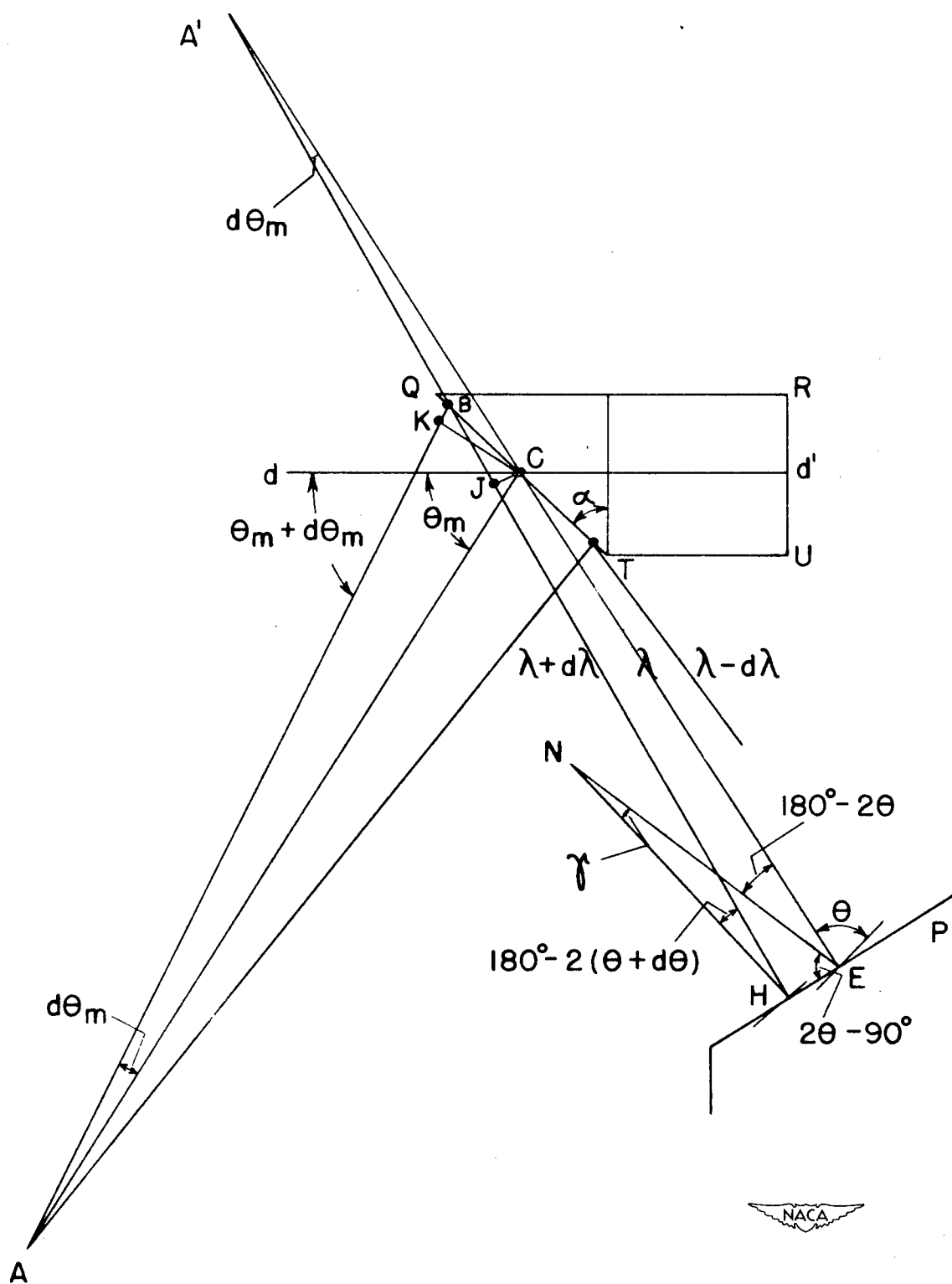


Figure 1.- Schematic diagram of X-ray achromatization with a point source.

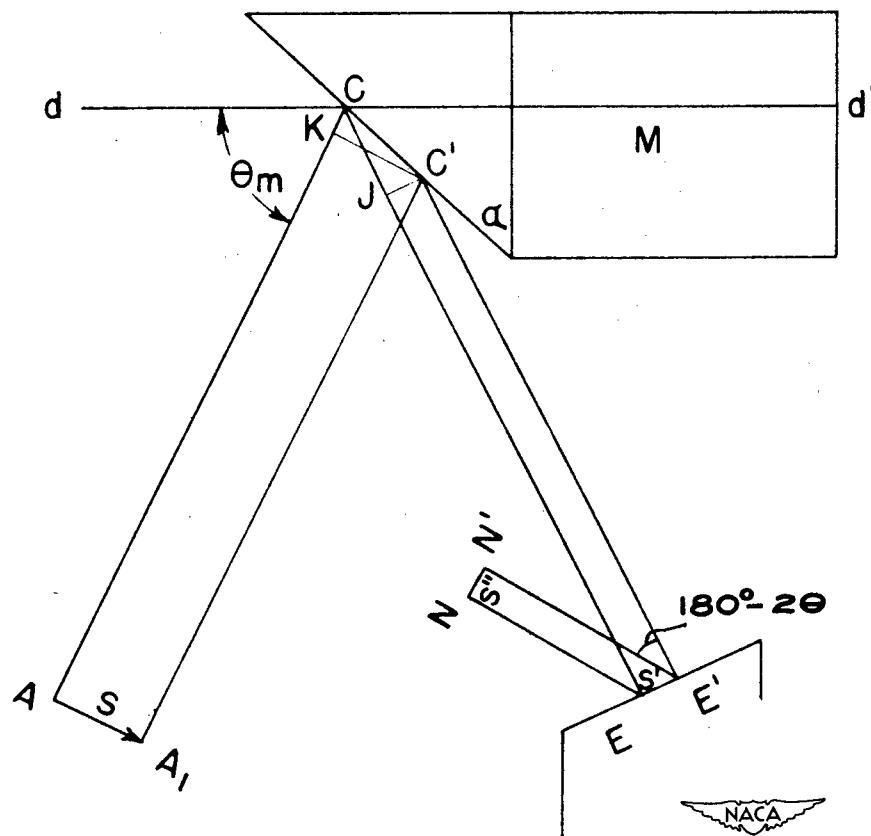


Figure 2.- Schematic diagram of X-ray achromatization with an extended source.

- J X-ray tube jacket
- M Monochromator
- A X-ray source
- W X-ray tube window
- ff' Film position
- N Image
- P Sample

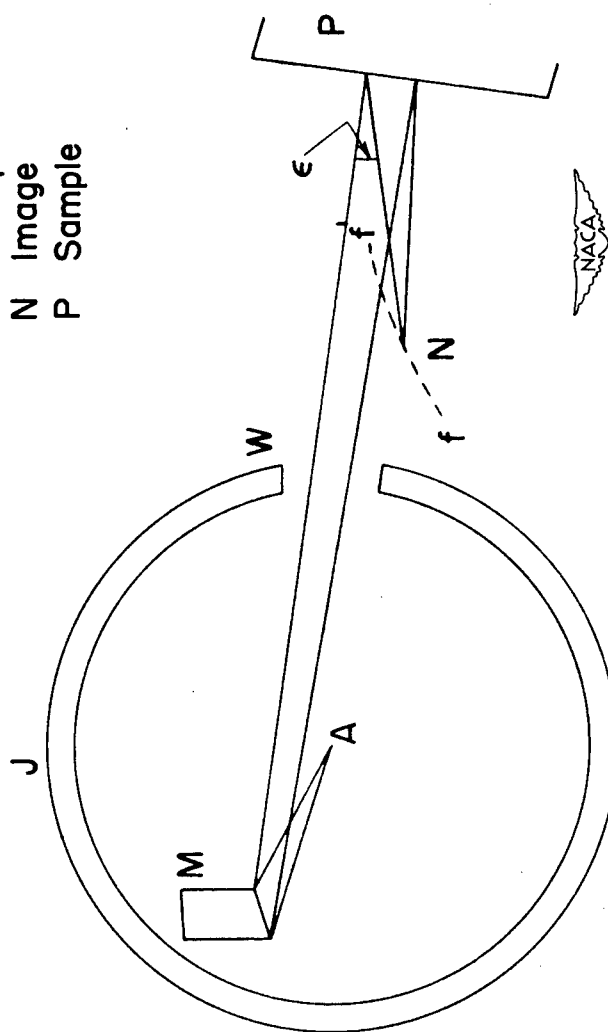


Figure 3.- Geometric arrangement of apparatus for X-ray achromatization.

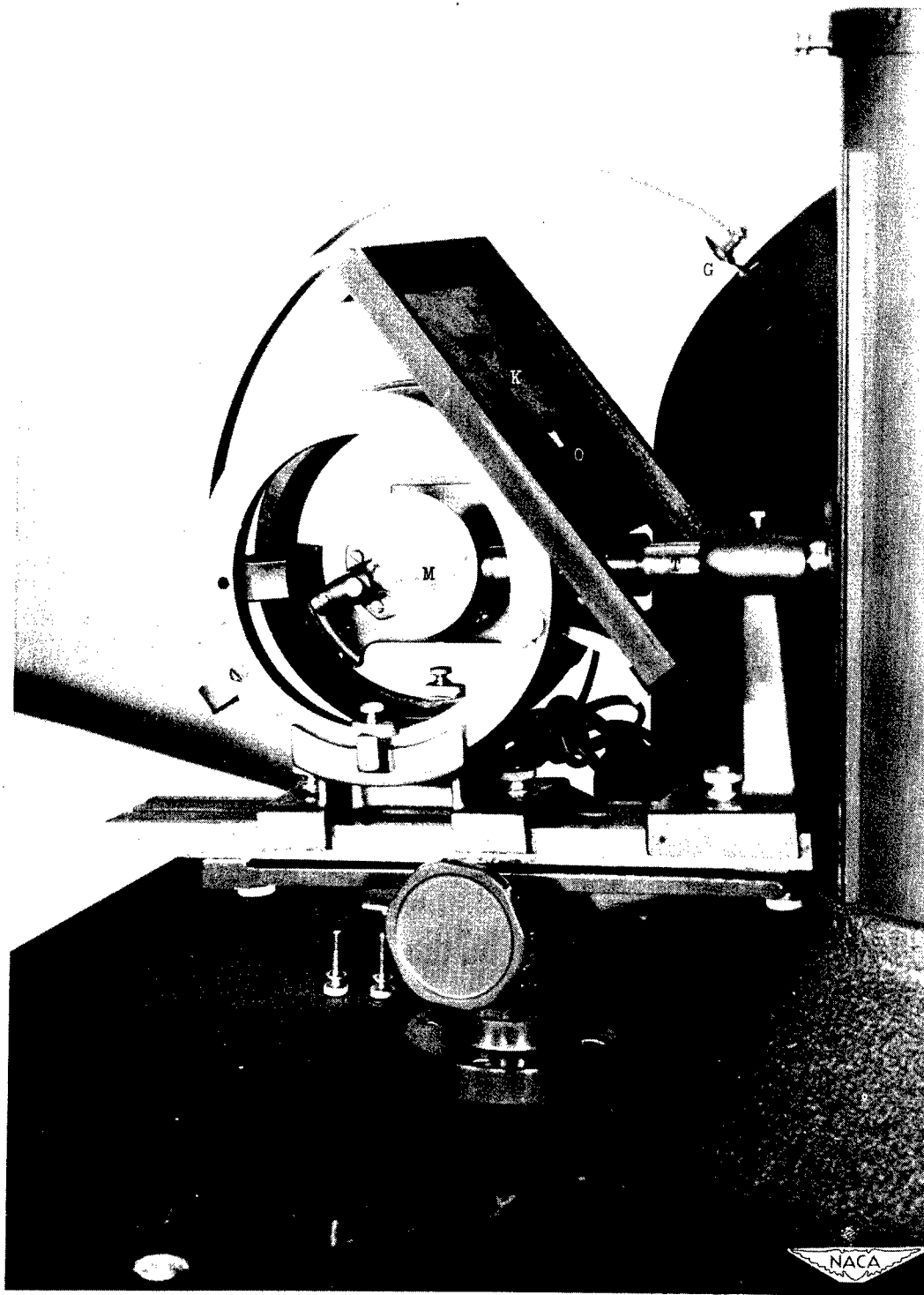
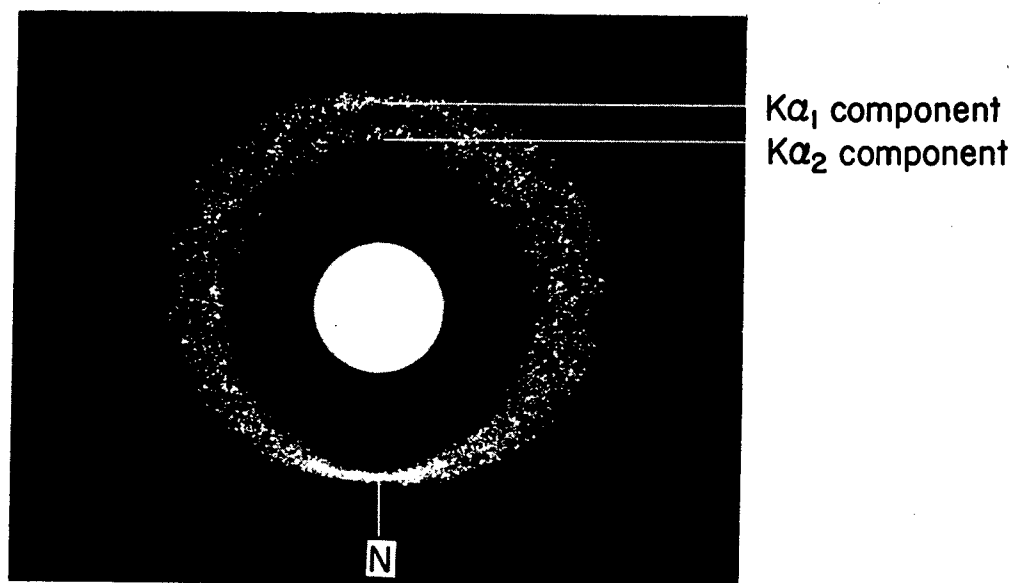
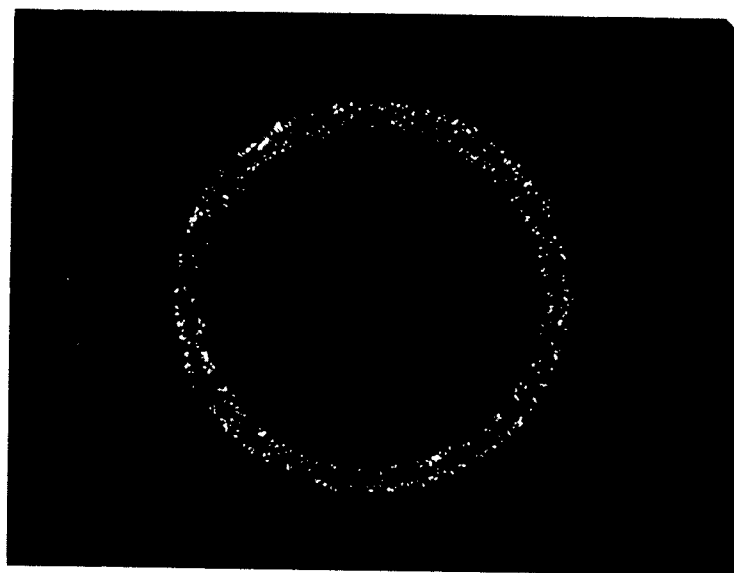


Figure 4.- Experimental arrangement of apparatus for X-ray achromatization.
G, specimen holder; K, film holder; O, aperture in film holder;
M, monochromator; T, X-ray collimator tube.



(a) Achromatized line.



(b) Debye-Scherrer photograph.



Figure 5.- Diffraction patterns of iron sample after 100-hour exposure. X-ray tube operated at 40 kilovolts and 7 milliamperes.

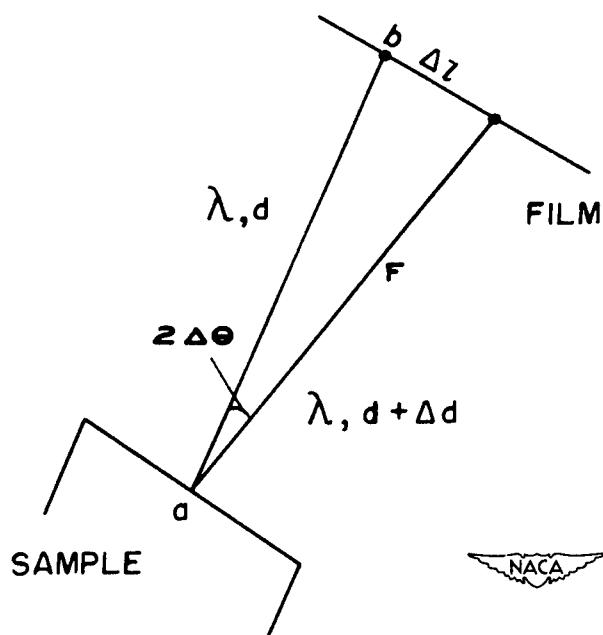


Figure 6.- Schematic diagram of diffracted rays.

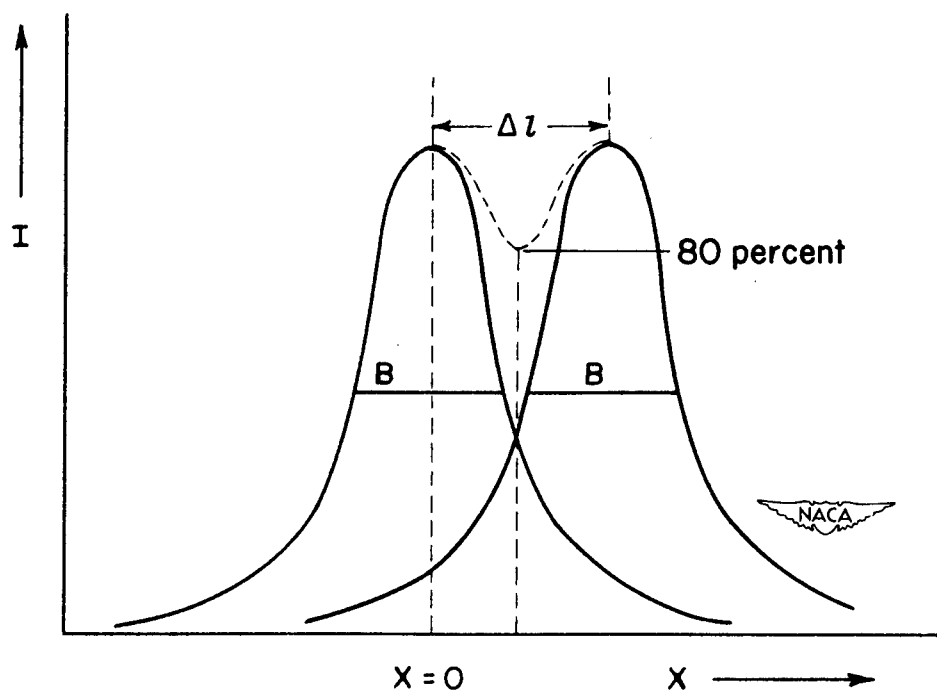


Figure 7.- Schematic diagram showing intensity distributions of two diffraction lines on the film.